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EFFECT OF LONG-TIME, ELEVATED-TEMPERATURE EXPOSURES TO VACUUM AND LITHIUM ON THE PROPERTIES OF A TANTALUM ALLOY, T-111

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16. Abstract The purpose of this study was to determine the effect of long-term, elevated-temperature vacuum and lithium exposures on the mechanical properties of T-111 (Ta-8W-2Hf). Exposure conditions were for 1000 hours at 980° or 1315° C, 5000 hours at 1315° C, and a duplex temperature exposure of 1000 hours at 980° C plus 4000 hours at 1040° C. The exposures resulted in reduced tensile and creep strengths of the T-111 in the 900° to 1100° C temperature range where a dynamic strain-age-strengthening mechanism is operative in this alloy. This strength reduction was attributed to the depletion of oxygen from solid solution in this alloy.					
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SUMMARY

The effect of long-term, high-temperature lithium and vacuum exposures on the tensile and creep properties of T-111 (Ta-8W-2Hf) were investigated in this study. The exposure conditions were for 1000 hours at 980⁰ or 1315⁰ C, 5000 hours at 1315⁰ C, and a duplex temperature exposure treatment of 1000 hours at 980⁰ C plus 4000 hours at 1040⁰ C. The purpose of these exposures was to simulate the environment expected in a liquid-metal-cooled nuclear reactor concept.

Both the lithium and the vacuum exposures reduced the tensile strength and the creep life of the T-111. The mechanism of strength reduction was thought to be associated primarily with a depletion of oxygen from solid solution, which reduced the effectiveness of a complex dynamic strain aging mechanism which produces significant strengthening of the T-111 in the 500⁰ to 1100⁰ C temperature range. The greatest oxygen depletions resulted from the lithium exposures.

Vacuum and lithium exposures at 1315⁰ C resulted in grain growth in the T-111. These exposures increased the creep life in the test temperature range where grain boundary sliding occurred and decreased the tensile elongation at the 1315⁰ C test temperature.

INTRODUCTION

A compact, lithium-cooled, fast spectrum space power reactor concept was studied at the NASA Lewis Research Center (ref. 1). The operating goal for the proposed reactor was 50 000 hours at a fuel element cladding temperature of 980⁰ C. The T-111 alloy

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(Ta-8W-2Hf) was chosen as a fuel element cladding and lithium containment material for this concept because of its good formability and low-temperature ductility and because of its desirable mechanical properties at the proposed reactor operating temperature. In addition, T-111 has shown promise for applications in the proposed lithium environment (ref. 2). It was not known, however, whether long-term exposure in a lithium environment at the reactor operating temperature would affect the mechanical properties of the T-111. The present study was undertaken to obtain such knowledge both for application to the present reactor concept and for further characterization of this strain-aging alloy from a metallurgical viewpoint.

In this study, T-111 sheet specimens were exposed to both lithium and vacuum environments for prescribed time and temperature conditions. These conditions were 1000 hours at 980°C or at 1315°C , 5000 hours at 1315°C , and a duplex temperature exposure of 1000 hours at 980°C followed immediately by 4000 hours at 1040°C . The lower exposure temperatures represented the proposed reactor operating temperature of 980°C , with allowance for temperature fluctuations to 1040°C . The 1315°C temperature was chosen as the maximum design operating temperature should greater reactor efficiency be desired by operating at higher fuel element cladding temperatures. The exposure times selected were reasonably long so that exposure effects should become apparent. After the exposures, bend tests, tensile tests, and creep tests were conducted to determine the effects of these exposures on the mechanical properties of the material. Microstructural, chemical, and X-ray analyses were used as aids in interpreting the data.

EXPERIMENTAL METHODS

Material

The material used in this study was commercially produced, 0.05-centimeter (0.020-in.) thick T-111 sheet. The actual amounts of tungsten and hafnium in this lot of T-111 were 7.9 and 1.95 weight percent, respectively. The interstitial analysis of the material is shown in table I. Bend, tensile, and creep test specimens were machined from this material to the configurations shown in figure 1.

The specimens were initially annealed for 1-hour at 1650°C in a vacuum of 5×10^{-5} newtons per square meter (4×10^{-7} torr) or better; this represents the standard recrystallization anneal for T-111 (ref. 2). A subsequent vacuum anneal of 1 hour at 1315°C was given the specimens. This anneal was used to simulate the postweld stress-relief treatment which would be applied to a fabricated hardware item.

Equipment and Procedures

Lithium and vacuum exposures. - Lithium and vacuum exposures were performed at a General Electric Company facility under a NASA contract (ref. 3). Specimens scheduled for exposure to lithium were sealed inside lithium-filled T-111 capsules. The lithium had an oxygen content of about 100 parts per million (ppm). The specimens which were to be exposed to vacuum were attached to the outer walls of the capsules to ensure that temperature conditions would be comparable to those of the lithium-exposed specimens. Four such capsules were prepared. Each capsule contained 11 tensile-type specimens and four bend specimens on the inside and a like number were attached to the outside of the capsules. Photographs of the capsules and specimens are shown in figure 2. The four capsules were heated to the proper exposure temperatures in an ion-pumped vacuum chamber containing four individually controlled tantalum heaters. Vacuums of about 10^{-7} newton per square meter (10^{-9} torr) were maintained at the test temperatures. Temperatures were monitored by tungsten - 3 percent rhenium tungsten - 25 percent rhenium thermocouples placed at the top, bottom, and center of each capsule.

After 1000 hours at temperature (980° or 1315° C), the heaters were turned off and the capsules were allowed to cool to room temperature. One of the 980° C capsules and one of the 1315° C capsules were removed. The remaining two capsules were heated for an additional 4000 hours at capsule temperatures of 1040° or 1315° C. Thus, the test specimens were exposed to the eight conditions described in table I. (The exposures involving 1000 hours at 980° C and 4000 hours at 1040° C are hereafter referred to as "duplex" exposures.)

At the completion of the exposure runs, the vacuum-exposed specimens were removed from the exterior of the capsules. The capsules then were cut open in an inert gas atmosphere chamber, and the lithium was heated and drained off. Subsequent cleaning in liquid ammonia and ethanol served to remove any residual lithium from the specimens.

After cleaning, the carbon, oxygen, nitrogen, and hydrogen contents were determined for a selected specimen from each group. The specimens were then packaged and shipped to NASA Lewis Research Center for evaluation.

Bend testing. - Room temperature bend tests were performed in air using selected specimens from each exposure condition. A punch-and-die type bend test fixture was used to bend the specimens through a 180° angle in three-point loading. The bend radius was 0.05 centimeter (0.020 in.), which was equal to the thickness of the specimen and thereby constituted a "1-t" bend radius. The apparatus was designed for use in a conventional tensile testing machine operating in the tensile mode. The crosshead movement rate was 2.5 centimeters per minute (1 in./min) for all bend tests. After bending, each specimen was further flattened upon itself, resulting in a "0-t" 180° bend.

Tensile testing. - Tensile specimens were prepared for testing by scribing gage marks into one of the flat surfaces at a 2.5-centimeter (1-in.) interval and measuring this distance to the nearest 0.013 centimeter (0.005 in.) to obtain the initial gage length for elongation determinations. The initial cross-sectional area of each specimen was determined by averaging several micrometer measurements of the thickness and width of the gage section.

Tensile testing was performed in a vacuum furnace using a commercially available screw-driven tensile testing machine. The diffusion-pumped vacuum system was capable of attaining vacuums of the order of 1.3×10^{-4} newton per square meter (1×10^{-6} torr) at temperature; however, during specimen heatup the pressure rose to about 1.3×10^{-3} newton per square meter (1×10^{-5} torr). The accuracy of temperature measurement was estimated to be about $\pm 3^{\circ}$ C. All tensile testing was done at a crosshead movement rate of 0.13 centimeter per minute (0.05 in./min).

After testing, the broken halves of the specimens were fit together, and the distance between gage marks was again measured for use in determining specimen elongation. (Reduction-in-area determinations were not made because the nature of the tensile fractures precluded obtaining meaningful posttest thickness and width measurements.) Part of the broken gage sections of selected specimens from each lot were examined by metallographic and chemical analytical (nitrogen and oxygen) procedures.

Creep testing. - Creep testing was performed at a TRW, Inc. facility under a NASA contract (ref. 4). The creep testing furnaces were ion-pumped and maintained at a vacuum in the 10^{-6} newton per square meter (10^{-8} torr) range during testing. Temperature was measured and controlled by two tungsten - 3 percent rhenium/tungsten - 25 percent rhenium thermocouples. A transistorized three-mode controller held the furnace temperature cycle to within about $\pm 1^{\circ}$ C during testing. Two additional thermocouples of the same type were used to monitor the temperature of the specimens in combination with a specially modified optical pyrometer having a temperature sensitivity of $\pm 0.6^{\circ}$ C. Uniaxial tensile loads of up to 48 meganewtons per square meter (70 ksi) could be applied to the specimens of this study by the use of stainless steel weights located inside the creep test units.

Creep extension was measured by means of a double-telescope, optical extensometer having an accuracy of 1.3×10^{-4} centimeter (5×10^{-5} in.). Creep readings were taken by sighting on two scribe marks located at a 2.54-centimeter (1-in.) interval on the surface of each test specimen. At the completion of a creep test, the furnace was cooled to room temperature with the specimen still under load.

The creep test temperatures and stresses used in this study were directed toward determining the 1-percent creep life in the 900° to 1200° C temperature range. These temperatures represent the operating range for the proposed nuclear reactor concept. Stresses were chosen at which 1-percent creep life could be obtained in a reasonable time (500 to 1000 hr) for the unexposed material which was tested in this study.

Supplementary stresses and temperatures were used as required to aid in characterizing the behavior of the material and in interpreting test results. Rupture data, as well as creep data, were obtained at 900° C.

RESULTS

Effect of Exposures on Composition and Microstructure

The effect of the exposures on the interstitial content and grain size of the T-111 is shown in table I. The original oxygen content for this material averaged about 30 ppm and remained near that level after the initial anneal and after most of the vacuum exposures. One exception was the 5000-hour, 1315° C vacuum-exposed material, which had only about 4 ppm oxygen after exposure. In contrast, the oxygen content of all the lithium-exposed specimens was lowered to the 3 to 12 ppm range. In all cases, the other interstitial contents remained about the same after exposure as they were before exposure - that is, 30 ppm for carbon, 6 to 14 ppm for nitrogen, and about 1 ppm for hydrogen.

The influence of exposure on the microstructure of the T-111 alloy is shown in figure 3. These photomicrographs show that the lower temperature exposures had no appreciable effect on grain size. But the high temperature (1315° C) exposures caused significant grain growth, with the grain size increasing from 0.028 millimeter to approximately 0.040 millimeter in 1000 hours (figs. 3(d) and (h)) and to over 0.1 millimeter in 5000 hours (figs. 3(e) and (i)). Approximately the same amount of grain growth occurred in both vacuum and lithium, indicating that the growth was not sensitive to environment.

The photomicrographs in figure 3 also show significant variations in the amount of precipitate which was present in some of the specimens. This precipitate was typical of that seen in numerous other heats of T-111 and has been previously identified as primarily a hafnium oxide (ref. 5). The short-term vacuum-exposed specimens showed essentially no change in precipitate concentration from the unexposed condition (figs. 3(b) and (d)), while the short-term lithium exposures caused small increases in the amount of precipitate (figs. 3(f) and (h)). However, the duplex exposures caused large increases in the amount of precipitate, with the vacuum exposure causing a larger increase than the lithium (figs. 3(c) and (g)). Both of the long-time high-temperature exposed specimens were essentially clean, with no precipitates visible at $\times 1000$ (figs. 3(e) and (i)). In no case was any surface degradation, pitting, grain boundary penetration, etc. observed for any of the specimens exposed in this study.

Bend Test Results

Bend tests performed in this study showed that, for all exposure conditions, the T-111 could be bent in air and flattened upon itself at room temperature without cracking. So none of the exposure conditions appeared to detrimentally affect the good room-temperature ductility of this material. (Bend tests were not performed at other temperatures because of the limited number of specimens.)

Tensile Test Results

Baseline data for annealed, unexposed T-111 specimens were obtained at several test temperatures such that the tensile properties could be characterized as a function of test temperature from 25⁰ (room temperature) to 1315⁰ C. The results are plotted in figure 4.

Figure 4 shows that both the ultimate and the yield strength of the unexposed material decreased with increasing test temperature up to about 500⁰ C, where a reversal in this trend occurred. A secondary peak in both ultimate and yield strength was observed at about 800⁰ C. At higher temperatures, the strength again decreased with increasing test temperature. This type of behavior in ultimate strength has been observed previously for T-111 and has been attributed to dynamic strain aging in the 500⁰ to 1100⁰ C temperature range (ref. 6).

The total elongation values for the material are also shown in figure 4. No abrupt changes in trend were observed for these data; the elongation decreased slightly with increasing test temperature (from about 30 percent at room temperature to about 18 percent at 760⁰ C) then increased to about 38 percent at the 1315⁰ C test temperature. Perhaps a better indication of the high ductility for this material was the observation that after testing most of the fractured sections of the specimens tested at temperatures below 1315⁰ C were drawn down to a point. Since this point could not be accurately measured to provide an absolute numerical value for reduction in area calculations, such data are not shown. However, values approaching 100 percent reduction in area were implied from the observations of these test specimens. The specimens tested at 1315⁰ C were more difficult to evaluate in this manner because of irregularities caused by grain boundary separation.

The exposed specimens, being fewer in number, were tested only at 25⁰, 900⁰, 980⁰, and 1315⁰ C. Data from these tests are compared with the baseline data in the several plots of figure 5. In most cases, the exposure conditions resulted in decreases in both the ultimate strength and the yield strength of the material at all test temperatures. These strength decreases varied with exposure and tensile test conditions, and they ranged up to a maximum of about a 17 percent decrease.

The strength values for the lithium-exposed specimens are, in general, slightly lower than those of comparable vacuum-exposed specimens. Also, the longer term (5000-hr) exposures generally caused a greater strength decrease than the 1000-hour exposures, regardless of the exposure temperature or environment (i.e., vacuum or lithium). The greatest decrease in strength resulted from the 5000-hour duplex exposure in lithium. This material had a 900°C ultimate tensile strength of 351 meganewtons per square meter (51 ksi) compared to 427 meganewtons per square meter (62 ksi) for the unexposed material. Also, the yield strength of this exposed material was lowered by a comparable degree (fig. 5(c)) at this test temperature. Comparative values for the yield strength over the entire temperature range of this study were difficult to obtain, as the shape of the tensile curve (load as a function of strain) underwent a transition in the 760° to 980°C range. At lower test temperatures, a drop-in-yield phenomena was observed, resulting in both upper and lower yield strength determinations. The lower yield strength was used to plot the data in this temperature range. At 1315°C , the tensile curves were uninterrupted or rounded off at the yield point; in these cases, the 0.2-percent offset yield strength was plotted. In the dynamic strain aging region, however, the drop-in-yield phenomena became less prominent, and the shape of the tensile curves underwent a transition from low-temperature to high-temperature behavior. In this region, the value appropriate for each specimen was used: the agreement between lower yield strength and a projected 0.2-percent offset yield strength in this region was very close, so that the yield strength relationship among the various specimens is as indicated on the plots of figures 5(c) and (d).

Contrary to the strength results, the exposure conditions had little effect on the ductility values for most tests. Elongation values for the exposed specimens were within a few percent of the baseline data except at the 1315°C test temperature, as is shown in figure 5(e). At 1315°C , these values ranged from 15 percent for material exposed to lithium for 5000 hours at 1315°C to 55 percent for the lithium duplex-exposed material. Observations of the fractured ends of the exposed specimens also indicated an implied high ductility comparable with that of the unexposed material, with the same exception that for the large-grained specimens tested at 1315°C grain boundary separations prevented meaningful deductions of this type.

Creep Test Results

The creep and rupture-life data obtained in this study are presented in table II. For ease of comparison, the life to 1-percent total creep strain for the unexposed and the exposed test specimens are shown in parametric (Larson-Miller) form in figure 6. In contrast to the tensile test results, the exposure temperature seemed to have the greatest effect on creep life. That is, large degradations in creep life were observed for the

lower temperature exposures while only small variations resulted from the 1315° C exposures.

The largest reductions in creep life were observed for the specimens which received the duplex exposure treatments. For example, the greatest reduction occurred in the lithium duplex-exposed specimen tested at 900° C and 345 meganewtons per square meter (50 ksi). This reduction in life to 1-percent total creep strain was from 938 hours to only 2 hours.

It is also apparent from the figure that for any given comparable exposure time and temperature the life to 1-percent creep strain for similarly tested specimens shows most of the lithium-exposed T-111 to be less creep resistant than the vacuum-exposed material. The only exceptions are for the 5000-hour, 1315° C exposed specimens tested at 1200° C and 110 meganewtons per square meter (16 ksi). At this test condition, the lithium-exposed material exhibited longer creep life than the vacuum-exposed material. And both of these specimens were stronger than similarly tested unexposed material.

The effect of the various exposure conditions on the shapes of the resulting creep curves can be observed for several test conditions in figure 7. Large variations in shape were observed as a result of these exposures and test conditions, as will be discussed later.

DISCUSSION

Interpretation of Results

The results of this study have shown that neither the room temperature bend ductility nor the outward physical appearance of the T-111 specimens of this study were affected by long-term, elevated-temperature exposures to either vacuum or lithium. However, the major effects which did result from these exposures were (1) the depletion of oxygen from the material resulting from all lithium exposures and one vacuum exposure, (2) the grain growth resulting from the highest temperature exposures, and (3) the changes in tensile strength and creep properties resulting from most of the exposures.

Of particular interest were the reductions in creep life which occurred at 900° and 980° C and the decreases in tensile strength also observed at and near these temperatures. It is significant to note that this temperature regime is included in a temperature range where a sensitivity to dynamic strain aging has been observed for the T-111 alloy (i. e. , 500° to 1100° C) (ref. 6). Therefore, interpretations of test results obtained in this temperature range should consider the effects of this phenomenon, especially as it is related to the interstitial content of the material (which was greatly affected by the long-term exposures of this study).

Effect of oxygen in solid solution. - Dynamic strain aging as it occurs in the T-111 alloy is assumed to be the result of a complex atmosphere dislocation interaction. The interstitial specie associated with this interaction in T-111 has been identified as oxygen (ref. 6). The strengthening effect results from a complex interaction between the oxygen and a highly reactive solute additive which involves preferential migration of the two solute species to the vicinity of dislocation cores. This interaction effectively pins the dislocations and hence strengthens the material. Since the amount of the reactive metal species (assumed to be hafnium) in T-111 is many times greater than that required to react stoichiometrically with all of the available oxygen, the effect of the dynamic strain age strengthening varies directly with the amount of oxygen in solid solution. Thus, it is the amount of oxygen in solid solution, and not the total oxygen content of the alloy, which must be related to the observed variations in strength in this alloy.

Because of the importance of the residual oxygen level in solid solution in the T-111 alloy, an effort was made to evaluate these levels in the exposed specimens. While no quantitative method could be found to measure oxygen in solution, it was possible to make qualitative estimates based on a correlation of the measured total oxygen content and the observed amount of hafnium oxide precipitate present in the microstructure. Each specimen was classified on this basis as having a "typical," "low," or "very low" level of residual oxygen in solid solution. A "typical" rating was assigned to specimens which exhibited an oxygen analysis typical of as-fabricated T-111 (i.e., 20 to 30 ppm) and showed very little precipitate in the microstructure (figs. 3(a), (b), and (d)). A "low" rating was assigned to specimens which were estimated to have between 1 and 10 ppm of residual oxygen in solid solution. Both of the long-term, high-temperature exposure conditions represented in figures 3(e) and (i) fall into this category, or the chemical analyses of these specimens fall in the 1 to 10 ppm range and essentially no precipitate is visible in the microstructure. The duplex-lithium-exposed specimen, which produced an oxygen analysis of 4 ppm and exhibited a significant amount of precipitate in the microstructure (fig. 3(g)), was considered to have a "very low" (<1 ppm) level of oxygen in solid solution. Also, the duplex-vacuum-exposed specimen, which had a total oxygen analysis of 30 ppm but contained an extensive amount of precipitate (fig. 3(c)), was classified in the "very low" range because it is assumed that most of the oxygen is in the hafnium oxide precipitates. Both of the 1000-hour lithium exposed specimens (figs. 3(f) and (h)), which have low oxygen analysis with less precipitate than the duplex lithium specimen, were classified in the "low" range. A summary of these classifications is presented in table III.

Effect of grain growth. - While effects of the exposures on the dynamic strain aging mechanism are believed to contribute significantly to the property variations observed in this study, the effect of grain growth resulting from the exposure conditions is also an important consideration in analyzing the test results. At test temperatures beyond the range at which the dynamic strain aging mechanism is operative, increased grain size

can increase the creep life of the material over that of finer grained material. Thus, the relatively large grain sizes observed for the specimens exposed at 1315°C are probably responsible for the relatively small changes in creep strength exhibited by these specimens (fig. 6(b)). In fact, the largest grained material had higher creep strength than the unexposed material when tested at 1200°C (table II). It is believed that the higher creep rate of the unexposed (relatively finer grained) material is due to the contribution of grain boundary sliding to total creep deformation at the higher test temperatures.

Effects on tensile properties. - The variations of tensile strength with the exposure conditions shown in figures 5(a) and (b) may be rationalized on the basis of both the observed grain growth and the loss of solid solution oxygen which occurred during exposure. In the temperature range where the dynamic strain aging effect was believed to be operative (500° to 1100°C), the observed strength variations correlated well with the estimated loss of oxygen from solid solution (table III). For example, the three specimens which were rated as having "typical" levels of oxygen in solid solution exhibited the lowest changes in tensile strength values, while the two specimens which were rated as having "very low" amounts of oxygen in solid solution were among the weakest specimens tested at 900° and at 980°C . Specimens which were rated as having "low" amounts of oxygen in solid solution had tensile strength values which were between the strengths of the specimens rated as "typical" or "very low."

At test temperatures outside the strain-aging-affected temperature range, the cause of the change in tensile strength was not clearly definable. At room temperature, essentially all of the exposed specimens were weaker than the unexposed specimens. However, the strength variations did not correlate well with the estimated oxygen levels of table III, except that the specimens having "typical" residual oxygen contents did have higher strengths than the specimens rated as having either "low" or "very low" residual oxygen levels. Also, the two specimens which exhibited the largest amount of grain growth also had the lowest tensile strength values. However, the two specimens which exhibited a small amount of grain growth (the 1000-hr, 1315°C vacuum and lithium-exposed specimens) were near the top of the scatter band in figure 5(b). Therefore, no clear correlation could be developed between strength and the observed effects of exposure on composition and structure.

The observed strength variations at the 1315°C tensile test temperature were even more difficult to interpret, since both reductions and increases in strength were observed as compared with the unexposed material. There did not appear to be any correlation between the estimated oxygen levels and the observed strength variations. As with the room temperature data, the two samples which exhibited large amounts of grain growth had the lowest strength values.

In general, the yield strength of the T-111 tested in this study (figs. 5(c) and (d)) was affected by the exposure conditions in the same manner as just discussed for the ultimate strength.

The effect of exposure conditions on the total tensile elongation of the material was apparent only at 1315° C (fig. 5(e)). A direct comparison of grain size (from fig. 3) with elongation is evident: the larger the grain size, the lower the elongation value. This relationship applies where tensile deformation is controlled by grain boundary sliding, which was the case for all the material tensile tested at 1315° C in this study. This was confirmed by metallographic observations of the specimens' fracture zones. Thus, exposure conditions which increased the grain size of the T-111 resulted in decreased total tensile elongation of the material.

Effects on creep properties. - As was the case for tensile results, the creep rate appears to be essentially a function of the residual oxygen left in solution in the material. Also, grain size effects were limited to the high-temperature exposure series since no grain growth occurred at the lower exposure temperatures. For the specimens exposed at 1315° C, both grain growth and deoxidation effects occurred, making it difficult to determine which had the greater effect on subsequent creep test results. Therefore, the grain size effect was evaluated at the 1200° C test temperature where this effect was expected to be the greatest and the effect of deoxidation on creep properties should be the smallest.

At high homologous temperatures, where grain boundary sliding contributes significantly to creep deformation (e.g., above about 0.45 T_m , or 1200° C for T-111), the creep rate is usually inversely proportional to the grain size. Garofalo (ref. 7) confirms this relationship for lead, tin, monel, and an Fe-Cr-Ni-Mn alloy tested at such temperatures. Since creep rate is inversely related to creep life, then the measured creep life at high homologous temperatures should be directly proportional to grain size. A plot of measured 1-percent creep life as a function of grain size is shown in figure 8 for the unexposed and the four high-temperature-exposed specimens tested at 1200° C. The plot has a slope of about 0.75, which indicates a significant contribution of grain boundary sliding as the creep-rate-controlling mechanism of both the oxygen-containing and the deoxidized T-111.

The influence of deoxidation on the creep behavior of T-111 was evaluated by an analysis of creep curve shape. The observed creep curves exhibited significant shape variations which depended on both exposure and creep test conditions. The changes resulting solely from variations in creep test conditions are illustrated in figure 9, where the variations of instantaneous creep rate with creep strain are plotted for the unexposed specimens. These plots were based on the conventional type of creep curve plots of figure 7, but they eliminated time as a variable axis. Thus, comparisons of creep curve shapes can be made on similar-size plots for test series which have widely varying time axes. The negative "spike" apparent in most of the plots indicates a slowing down of creep rate with time at low percentages of total strain; this is comparable to the "first stage" of creep on a conventional type of creep curve. A low value of a "spike" indicates a severe slowing down of creep rate, or nearly a cessation of the creep rate. This would

represent a horizontal portion on a conventional plot. It should be pointed out here that at very low instantaneous creep rates many hours passed with no observable effect on total creep strain. For example, the "cessation" of creep which occurred in figure 9(a) lasted for 92 hours, with no observable change in total creep strain. Less drastic decreases in instantaneous creep rate (fig. 9(e)) indicate that observable flow is continuously taking place, and no stoppage of accumulated creep strain is indicated. At the lowest creep test temperatures (900° and 980° C), where the dynamic aging phenomenon was most pronounced, the curves of figure 9 exhibited an unusual shape characterized by a complete cessation of creep over a finite period at a low strain level, followed by a resumption of creep with a steadily increasing creep rate (figs. 9(a) and (b)). Previous studies (ref. 6) show that the creep cessation in T-111 was associated with the dynamic strain aging phenomenon, and that the increase in creep rate subsequent to cessation was associated with vacuum-induced deoxidation which depleted the interstitial specie associated with the dynamic aging. It was also observed in reference 6 that after deoxidation the creep rate as a function of strain curve eventually became level over a finite strain interval, indicating the establishment of a steady-state creep rate which was characteristic of the deoxidized alloy. This phenomenon did not occur in the present tests. This is probably a result of the fact that the stress levels employed in this program were much higher than those used in the referenced study.

The creep cessation phenomenon did not occur at the 1095° and 1200° C creep test temperatures (figs. 9(c) and (d)). However, these curves did exhibit basically the same general shape as the lower temperature curves, indicating that the dynamic aging phenomenon may continue to play a role in the creep deformation of the T-111 alloy at these temperatures. At the highest test temperature used in this study (1315° C), the T-111 creep curve exhibited what might be considered a "normal" shape (fig. 9(e)).

An analysis of the creep curves for the exposed specimens showed that many of the exposures caused substantial reduction or complete elimination of the creep cessation phenomenon which occurred in the unexposed material at the lower test temperatures (fig. 7). This reduction in the effect of dynamic aging on creep was assumed to be associated with the deoxidation caused by exposure. To document this association, the shapes of the creep curves obtained at 900° C (where the dynamic aging effect was most prominent) were examined in more detail on plots of instantaneous creep rate as a function of total creep strain (fig. 10). These curves showed that the only exposures which completely eliminated the dynamic aging effect were the two duplex exposures to lithium and vacuum (figs. 10(c) and (g)). This observation may at first seem anomalous in the case of the duplex vacuum exposure, where chemical analysis showed a total oxygen level of about 30 ppm. However, it is consistent with the "very low" rating previously ascribed to the solid-solution oxygen levels in these samples because of the large amount of precipitates observed. It would thus appear that the duplex vacuum specimen has been deoxidized internally (in the sense of removing all of the oxygen from solid solution) by

the precipitation of hafnium oxide. The two 1000-hour vacuum exposures, where substantial oxygen appeared to have remained in solid solution, continued to display the creep cessation phenomenon (figs. 10(a) and (b)). The specimens from the remaining four exposure conditions did not show a creep cessation, but they did continue to exhibit slight dynamic aging effects, despite the low oxygen levels (figs. 10(d), (e), (f), and (h)). This observation indicates that the dynamic strain aging in this alloy requires only a few ppm of residual oxygen in solution to be operative.

Applicability of Results

Long-time elevated temperature exposures to either vacuum or lithium environments have resulted in a dramatic decrease in tensile strength and creep life of the tantalum alloy T-111. These decreases occurred in the operating temperature range of a conceptual advanced power reactor in which liquid lithium is intended to be the coolant and T-111 the fuel element cladding material. It is imperative, then, that these property changes be taken into account when design criteria are being established for this or any future reactor concept where T-111 will be exposed to either vacuum or liquid lithium.

Exposures of T-111 to vacuum and lithium were performed in this study as a method of simulating the conditions (except for irradiation) to which the material would be subjected when used as fuel element cladding or lithium containment material in an advanced lithium-cooled nuclear reactor concept. Although the initial reactor concept requires an operational lifetime of 50 000 hours, the 1000- and 5000-hour exposures of this study were used because of practical time consideration in performing the study, and because it was felt that effects resulting from such environmental exposures should be evident within these time periods. Furthermore, and perhaps most important in relating the data to the intended application, these two exposure times offer the opportunity to determine the effect of reactor operating time on the behavior of the material. For example, a decrease from 528 to 87 hours was observed in the life to 1-percent total creep strain for T-111 that had been exposed to lithium at 980°C for 1000 hours then tested at 980°C using an applied stress of 241 meganewtons per square meter (35 ksi). An exposure time of 5000 hours at nearly the same temperature (e.g., the duplex-exposed material) resulted in a 1-percent creep life of only 5 hours under the same test conditions. Similarly, a decrease in tensile strength with increased exposure time was observed (fig. 5) for this material at the 980°C test temperature. These results indicate that both the tensile strength and creep life decreased with increased exposure time at or near the intended reactor operating temperature (980°C). Also, for the indicated drastic reduction in creep life (528 to 87 hr) to occur after only 1000 hours of exposure time, the degradation process must have begun much earlier in the exposure cycle than 1000 hours. Since the life to 1-percent creep strain decreased to only 5 hours after a 5000-hour exposure, fur-

ther degradation would be expected at times in excess of the 5000 hours investigated in this study, or until such time as the mechanism responsible for the strength degradation becomes inoperative. Thus, since strength degradations would be expected early in reactor life and should worsen with increased operational time at 980°C , design-allowable properties should be based on conservative (low) values of strength and creep life obtained by thorough investigation of exposure effects, rather than on data available for unexposed T-111.

The case for increasing the power output potential of the proposed reactor by increasing the fuel element operating temperature was represented by the 1315°C exposure and test conditions. Creep tests at this operating temperature indicated an enhancement of strength at high temperatures. However, as with the low-temperature exposed material, degradation was also observed for the high-temperature exposed material which was tested at lower test temperatures. This degradation was also observed in tensile strength. This situation may be applicable during reactor cool-down, where the cladding temperature would pass through this lower temperature range at stresses imposed on it by the contained fuel which would be swelling because of irradiation effects. This situation could cause straining of the cladding material beyond its designed creep limitation.

The exposure and test conditions of this study can, in a sense, be related directly to the reactor design criteria proposed in reference 1. A limit of 1-percent total diametral fuel element cladding strain was established at both 980° and 1315°C operating temperatures, corresponding to the creep test criteria of this study. In this concept, the nuclear fuel must first expand enough to close the assembly gaps allowed between it and the cladding before stresses could be applied to the cladding. This contact of fuel and cladding is not expected to occur until several thousand hours of reactor operation have elapsed. This operating condition results in long-term exposure of the cladding material to lithium prior to the appreciable stressing of the cladding, similar to the long-term lithium exposure and subsequent creep testing done in this study. Thus, the results of this study can be considered indicative of the effects of exposure on the mechanical properties investigated. Therefore, qualitative estimates of strength and creep life may be obtained for design consideration from this study. However, much more extensive testing of this type is needed before quantitative design allowable properties can be established. In particular, long-term creep tests in a lithium environment are needed to evaluate the material in a stressed condition simultaneously with degradation or enhancement of creep strength. This would simulate the case during later stages in operation of the fuel element cladding and during the entire operation of lithium containment vessels and piping. Under these conditions, it is conceivable that the creep-life degradations would not be as great as those observed in this study.

SUMMARY OF RESULTS

This study has shown that exposures of T-111 (Ta-8W-2Hf) in vacuum or lithium environments for long times (1000 and 5000 hr) at elevated temperatures (980° or 1315° C, plus a duplex-temperature exposure of 1000 hr at 980° C plus 4000 hr at 1040° C) generally resulted in decreases in both the tensile and creep strengths of the material. The results of this study must be taken into account when considering T-111 for use in lithium environments in nuclear reactor systems, or in other applications where deoxidation or redistribution of oxygen within the material could be expected.

The following results summarize the major findings of this study.

1. The life to 1-percent creep strain for the T-111 tested in this study was reduced greatly in the 900° to 1100° C test range as a result of the exposure conditions. The greatest reductions resulted from the long-term duplex temperature exposures in either vacuum or lithium.

2. Reductions in tensile strength were observed from room temperature to 1315° C as a result of most of the exposure conditions. This reduction varied with exposure conditions up to a maximum of 17 percent loss in ultimate strength. Generally, the long-term exposures in lithium were most detrimental.

3. These reductions in strength are believed to occur primarily because of the effect of the exposure conditions on the loss of oxygen from solid solution which is involved in a dynamic strain aging mechanism in T-111. This deoxidation effect is attributed to loss to the exposure atmosphere (lithium or vacuum) or to precipitation of hafnium oxides.

4. Exposures to vacuum or lithium at 1315° C resulted in grain growth in the T-111. These exposures increased the creep life in the test temperature range where grain boundary sliding occurred and decreased the tensile elongation at the 1315° C test temperature.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, September 11, 1973,
503-25.

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Rep. GESP-189, General Electric Co. (NASA CR-72505), Mar. 20, 1969.
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TABLE I. - CHEMICAL ANALYSIS AND GRAIN SIZE OF T-111 SHEET EXPOSED TO VACUUM AND LITHIUM

Exposure condition	Average concentration, ^a ppm by weight				Average grain size, ^b mm
	Oxygen	Nitrogen	Hydrogen	Carbon	
As-received and pre-exposure annealed 1 hour at 1650° C + 1 hour at 1315° C	30	6	1	33	0.028
Exposed to 10 ⁻⁷ newton per square meter (10 ⁻⁹ torr) vacuum:					
1000 Hours at 980° C	24	6	1	36	.027
1000 Hours at 1315° C	29	6	1	36	.039
1000 Hours at 980° C plus 4000 hours at 1040° C (duplex vacuum)	30	7	1	26	.026
5000 Hours at 1315° C	4	14	1	24	.110
Exposed to lithium:					
1000 Hours at 980° C	12	6	1	25	.027
1000 Hours at 1315° C	3	6	1	38	.043
1000 Hours at 980° C plus 4000 hours at 1040° C (duplex lithium)	4	7	1	29	.028
5000 Hours at 1315° C	6	7	1	21	.119

^aCarbon determined by combustion; oxygen, nitrogen, and hydrogen determined by fusion method.^bGrain size determined by circle-intercept method.TABLE II. - EFFECT OF LONG-TERM VACUUM AND LITHIUM EXPOSURES
ON CREEP AND RUPTURE PROPERTIES OF T-111 SHEET

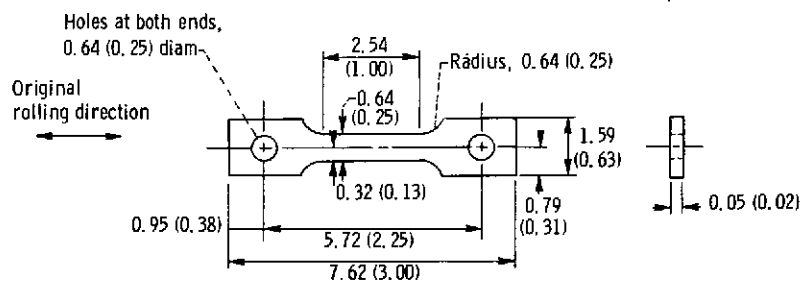
Creep test conditions	Unexposed	Vacuum exposures				Lithium exposures			
		1000 hr at 980 ⁰ C	1000 hr at 1315 ⁰ C	Duplex ^a	5000 hr at 1315 ⁰ C	1000 hr at 980 ⁰ C	1000 hr at 1315 ⁰ C	Duplex ^a	5000 hr at 1315 ⁰ C
	Life to 1-percent total creep strain, hr								
900 ⁰ C and 345 MN/m ² (50 ksi)	938	262	1278	4	340	43	448	2	180
980 ⁰ C and 241 MN/m ² (35 ksi)	528	227	465	10	745	87	323	5	350
1095 ⁰ C and 165 MN/m ² (24 ksi)	410	123	215	55	205	65	91	10	200
1095 ⁰ C and 110 MN/m ² (16 ksi)	435	429	546	155	885	110	440	73	1100
1200 ⁰ C and 110 MN/m ² (16 ksi)	----	---	---	2780	---	---	---	1530	----
1200 ⁰ C and 72 MN/m ² (10.5 ksi)	2330	---	---	1475	---	---	---	732	----
1315 ⁰ C and 35 MN/m ² (5 ksi)	600	---	---	570	---	---	---	463	----
Rupture life, hr									
900 ⁰ C and 345 MN/m ² (50 ksi)	1242	420	1886	48	655	165	700	34	204
Rupture elongation, percent									
900 ⁰ C and 345 MN/m ² (50 ksi)	20.8	25.8	27.0	30.0	13.8	25.3	17.9	26.6	19.6

^aDuplex refers to 1000 hours at 980° C plus 4000 hours at 1040° C.

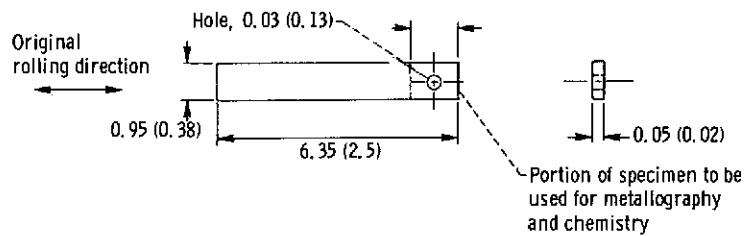
TABLE III. - QUALITATIVE RATING OF OXYGEN IN SOLID
SOLUTION FOR T-111 TEST SPECIMENS OF LONG-
TERM VACUUM AND LITHIUM EXPOSURE STUDY

[Oxygen level ratings: typical, 20 to 30 ppm; low, 1 to 10 ppm; very low, <1 ppm. Unexposed specimens, typical.]

Exposure environment	1000 hr at 980° C	Duplex	1000 hr at 1315° C	5000 hr at 1315° C
	Oxygen level rating			
Vacuum	Typical	Very low	Typical	Low
Lithium	Low	Very low	Low	Low

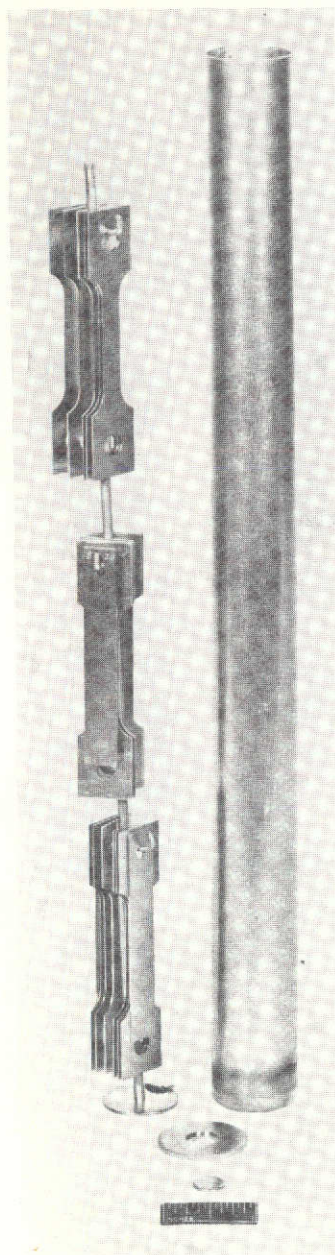


(a) Tensile and creep test specimen.

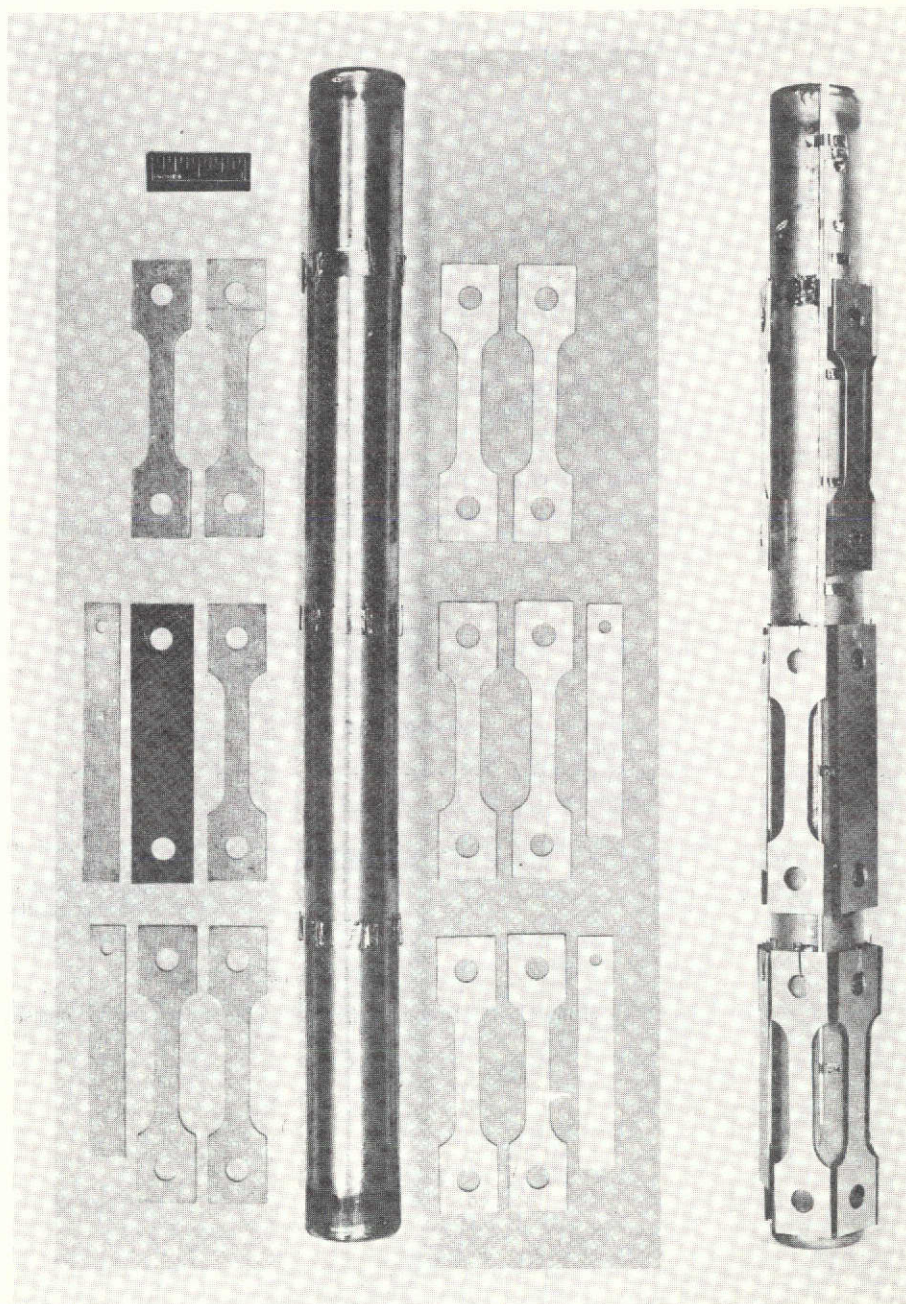


(b) Bend test specimen.

Figure 1. - T-111 sheet test specimens. Dimensions are in centimeters (in.).

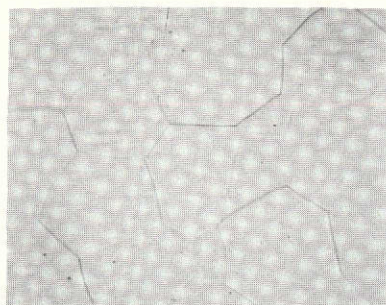


(a) T-111 specimens and capsule illustrating method used to mount specimens inside capsule.

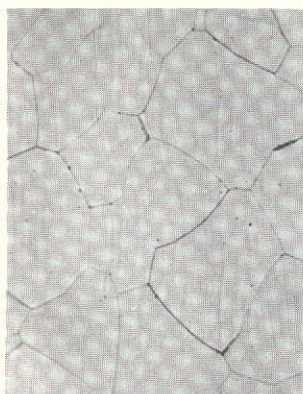


(b) Placement of specimens on outside of capsule.

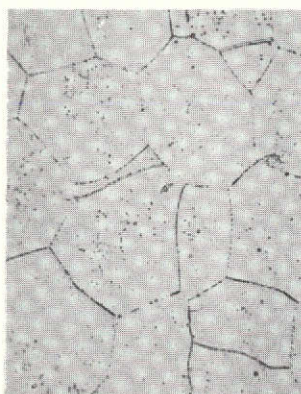
Figure 2. - T-111 specimens and capsules used in long-term exposure study.



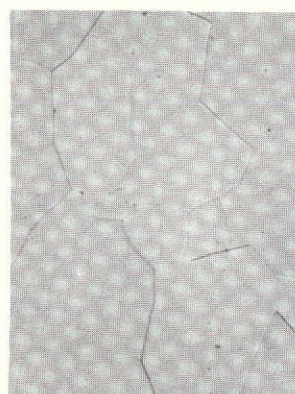
(a) Unexposed. Grain size, 0.028 millimeter; oxygen analysis, 30 ppm.



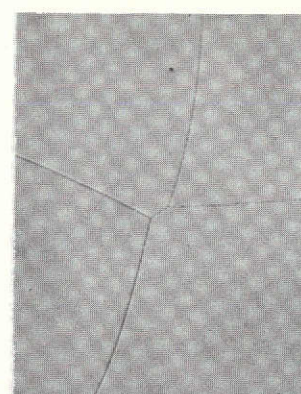
(b) Vacuum exposed, 980° C for 1000 hours. Grain size, 0.027 millimeter; oxygen analysis, 24 ppm.



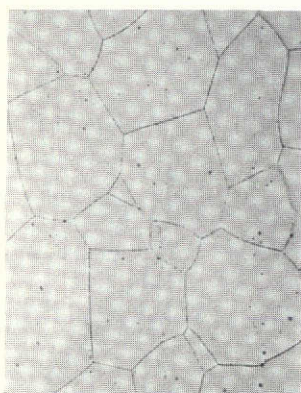
(c) Vacuum exposed, duplex. Grain size, 0.026 millimeter; oxygen analysis, 30 ppm.



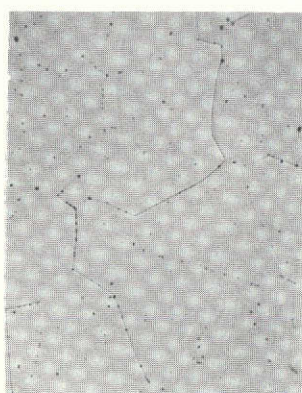
(d) Vacuum exposed, 1315° C for 1000 hours. Grain size, 0.039 millimeter; oxygen analysis, 29 ppm.



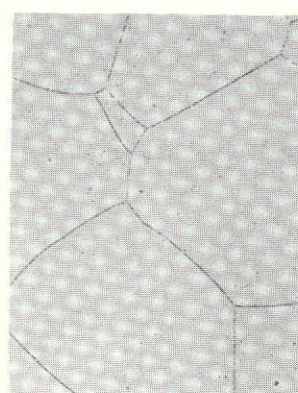
(e) Vacuum exposed, 1315° C for 5000 hours. Grain size, 0.110 millimeter; oxygen analysis, 4 ppm.



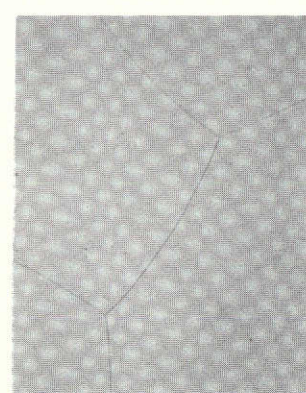
(f) Lithium exposed, 980° C for 1000 hours. Grain size, 0.027 millimeter; oxygen analysis, 12 ppm.



(g) Lithium exposed, duplex. Grain size, 0.028 millimeter; oxygen analysis, 4 ppm.



(h) Lithium exposed, 1315° C for 1000 hours. Grain size, 0.043 millimeter; oxygen analysis, 3 ppm.



(i) Lithium exposed, 1315° C for 5000 hours. Grain size, 0.119 millimeter; oxygen analysis, 6 ppm.

Figure 3. - Effect of long-time, elevated temperature exposure to 10^{-7} newton per square meter (10^{-9} torr) vacuum and to lithium on microstructure of T-111. All specimens annealed at 1650° C for 1 hour plus 1315° C for 1 hour in vacuum prior to exposure. Etchant: 50 cubic centimeters nitric acid, 20 cubic centimeters water, 30 grams ammonium bifluoride.

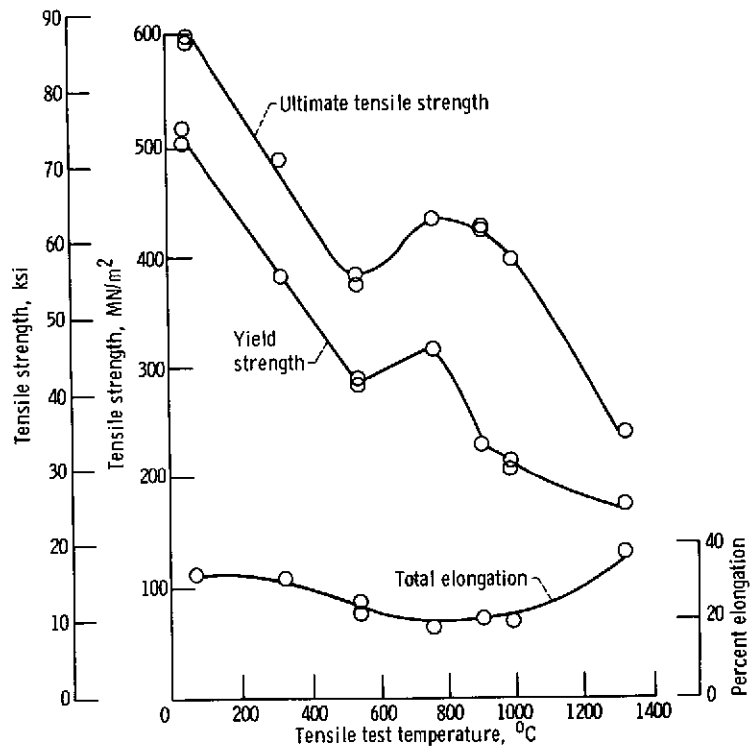


Figure 4. - Effect of test temperature on tensile properties of unexposed T-111.

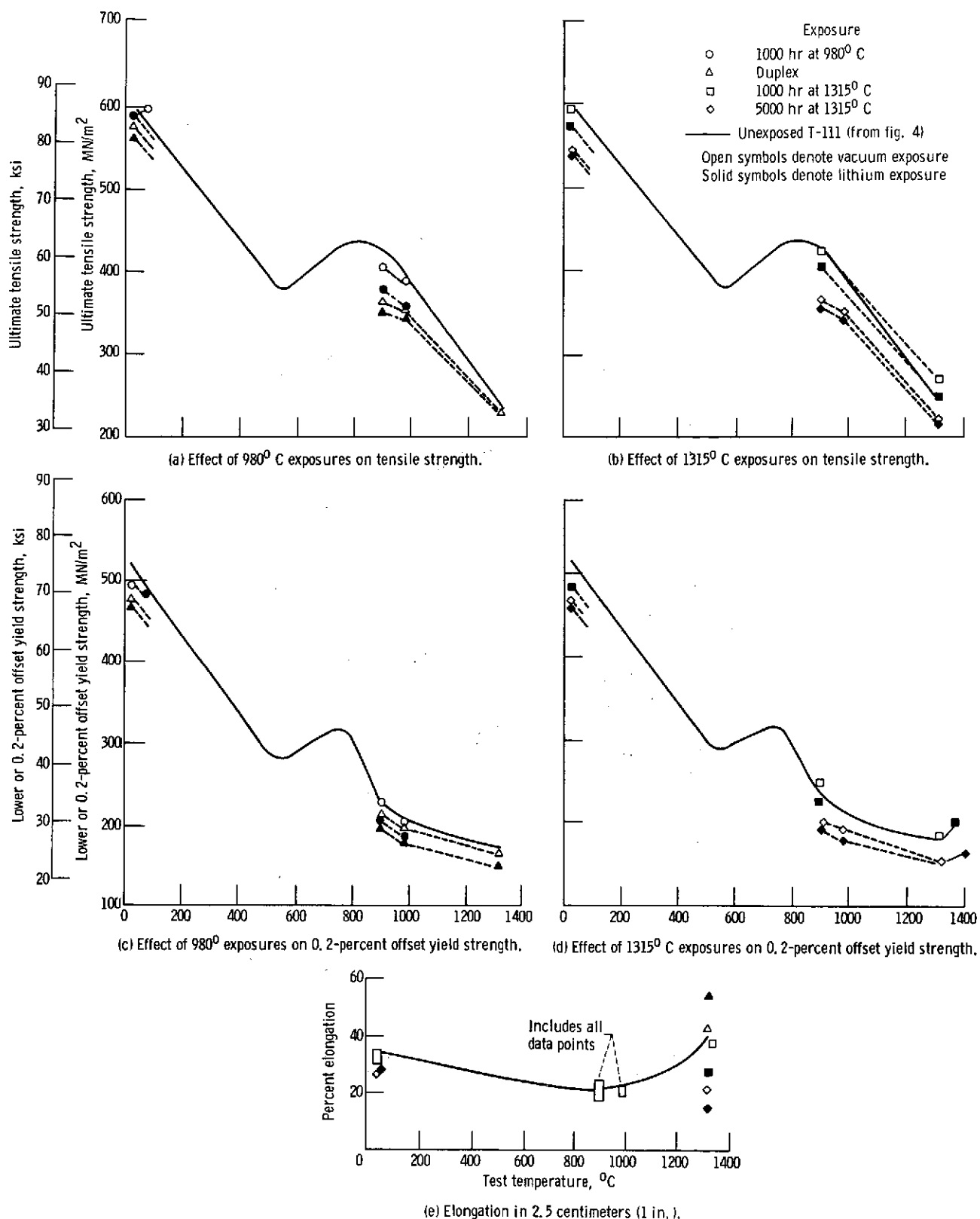


Figure 5. - Effect of long-term vacuum and lithium exposures on tensile properties of T-111 as function of test temperature. Duplex, 1000 hours at 980° C plus 4000 hours at 1040° C.

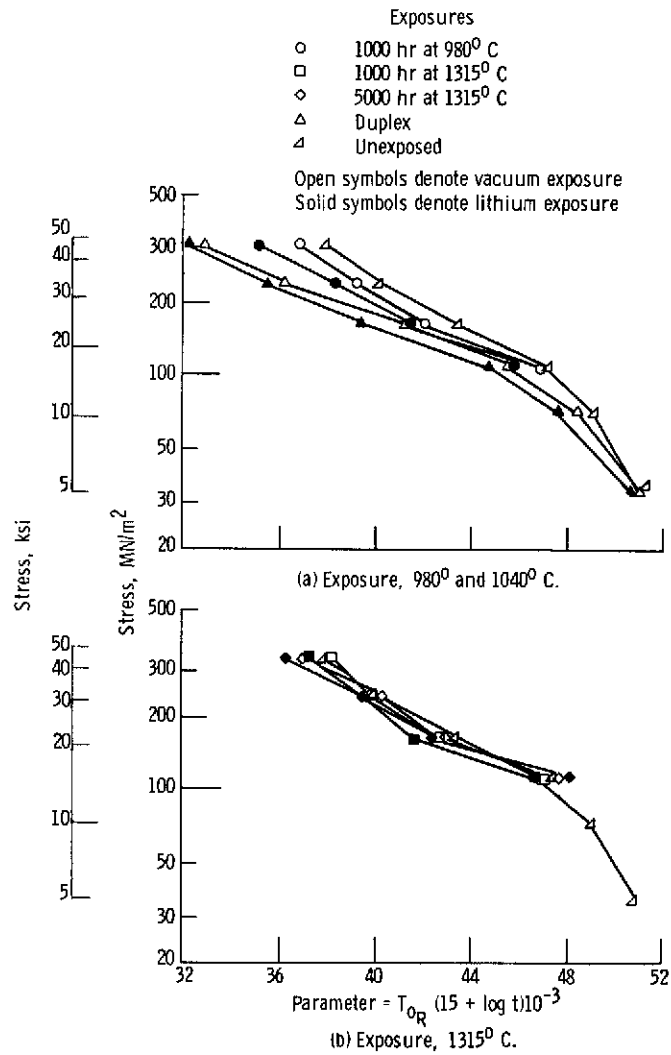


Figure 6. - Influence of long-time elevated-temperature exposure to lithium and 10^{-7} newton per square centimeter (10^{-9} torr) vacuum on life to 1-percent total creep strain of T-111 alloy.

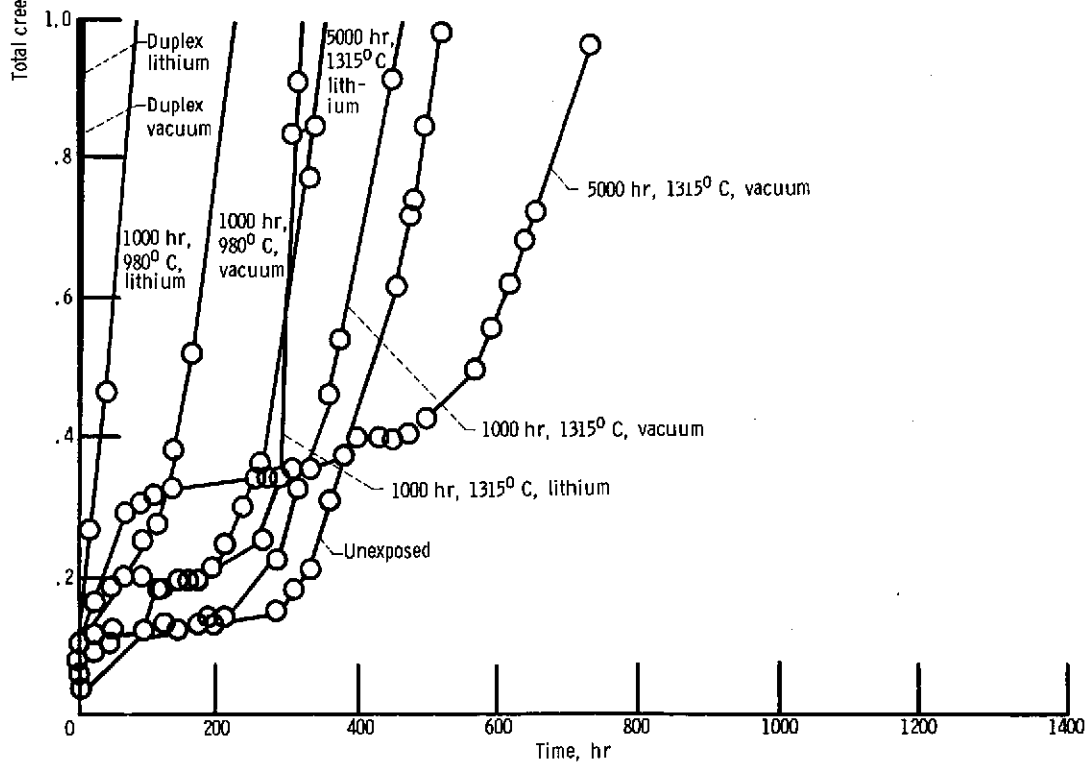
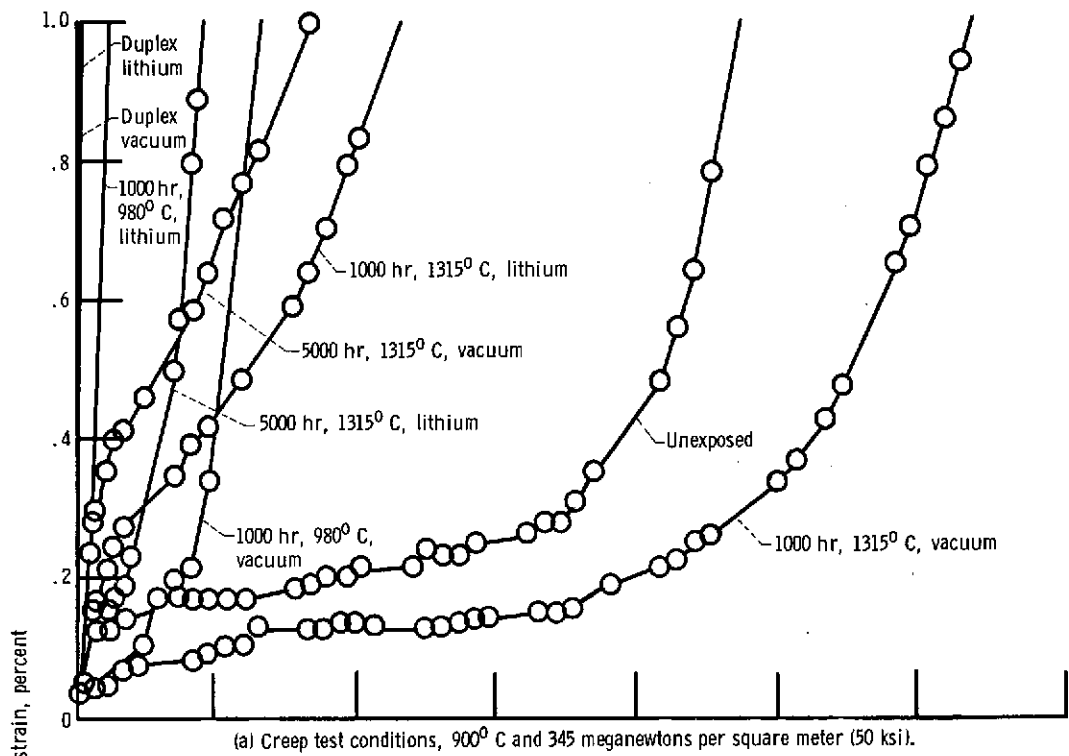


Figure 7. - Effect of long-term, high-temperature vacuum and lithium exposures on creep properties of T-111. Exposure conditions indicated for each creep curve. All creep tests in 10^{-6} newton per square meter (10^{-8} torr) vacuum range at test conditions indicated.

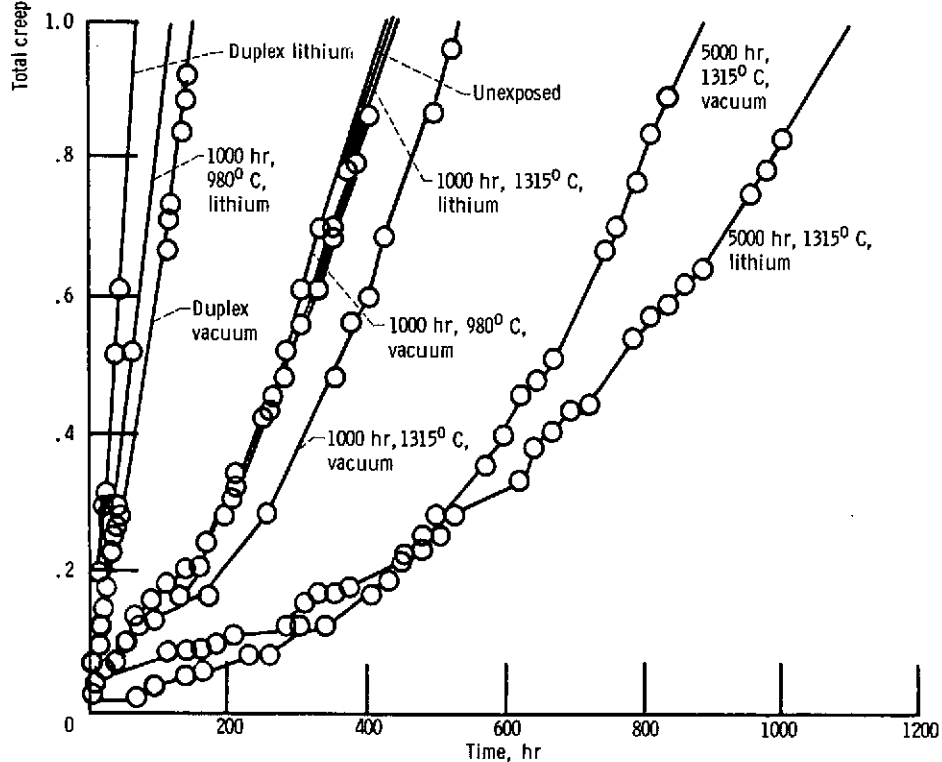
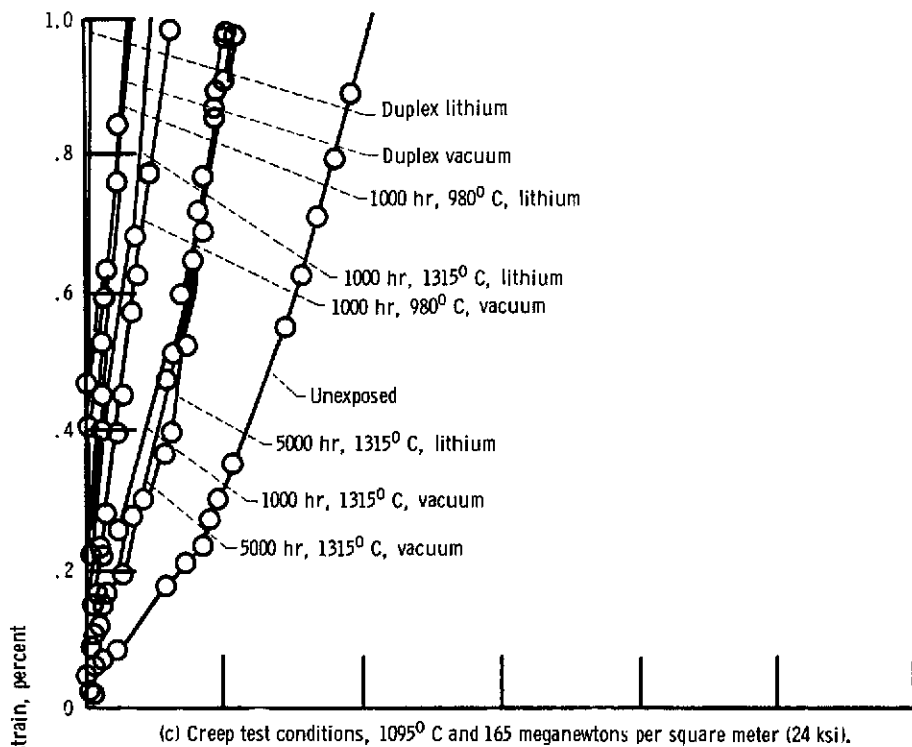
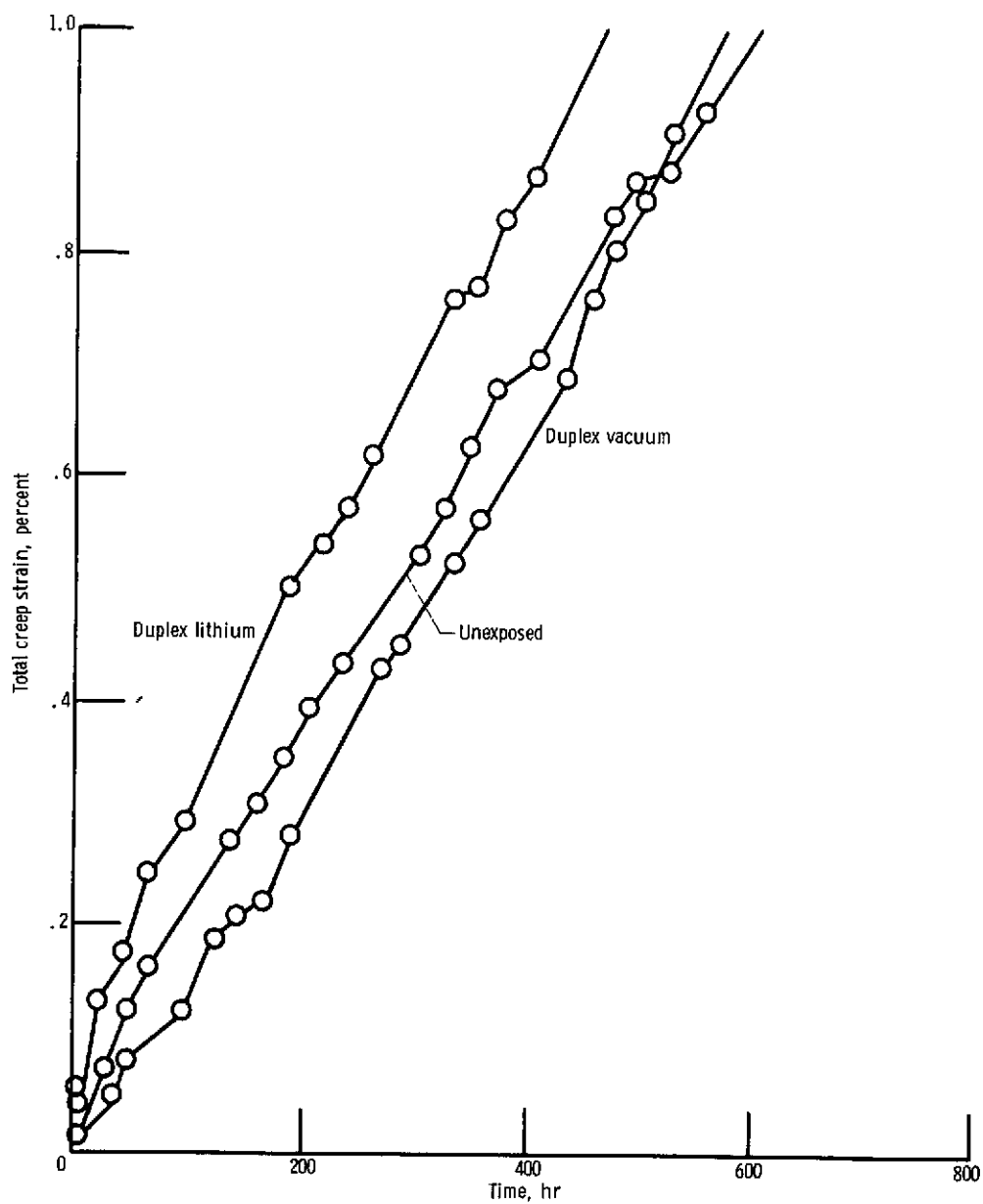


Figure 7. - Continued.



(e) Creep test conditions, 1315⁰ C and 35 meganewtons per square meter (5 ksi).

Figure 7. - Concluded.

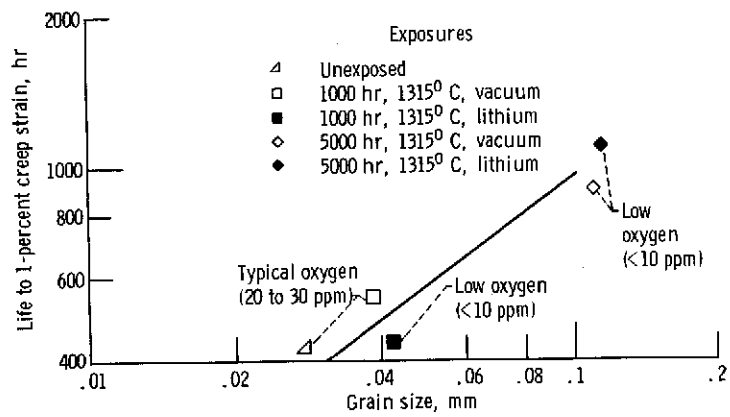


Figure 8. - Influence of grain size on life to 1-percent total creep strain of T-111 alloy exposed to lithium or vacuum at 1315° C for indicated periods. Creep test conditions, 1200° C and 110 meganewtons per square meter (16 ksi).

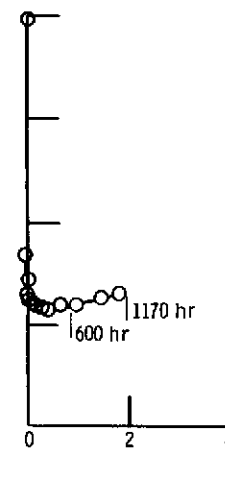
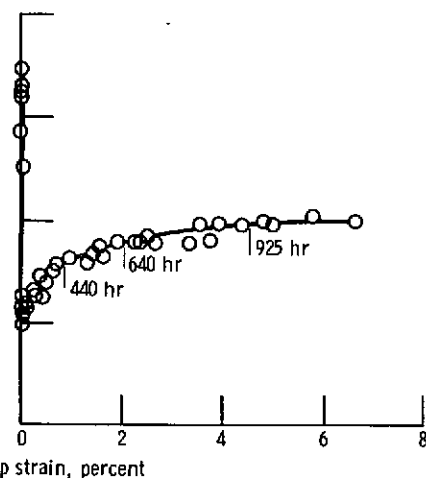
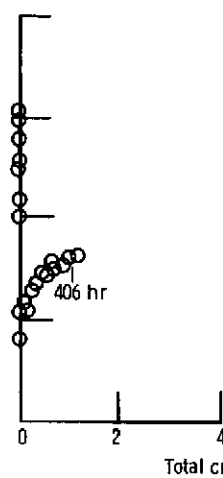
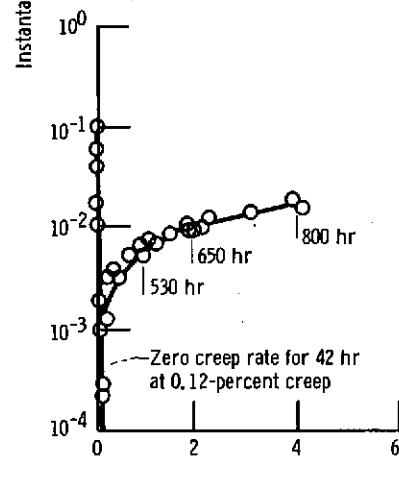
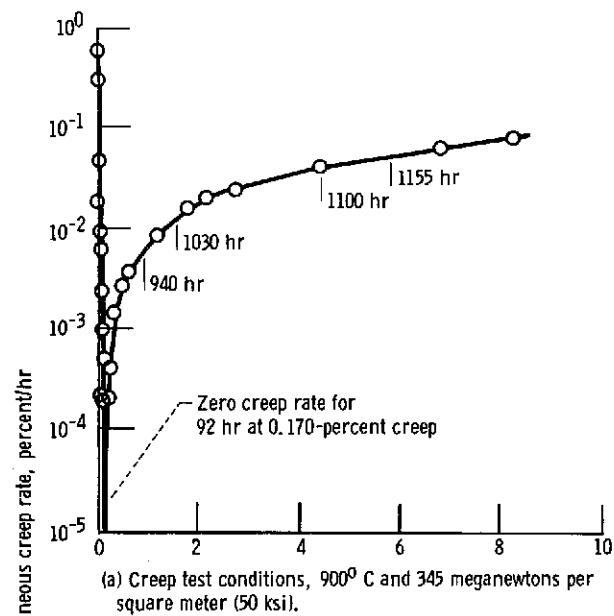


Figure 9. - Effect of creep test conditions on creep curve shape for unexposed T-111 alloy.

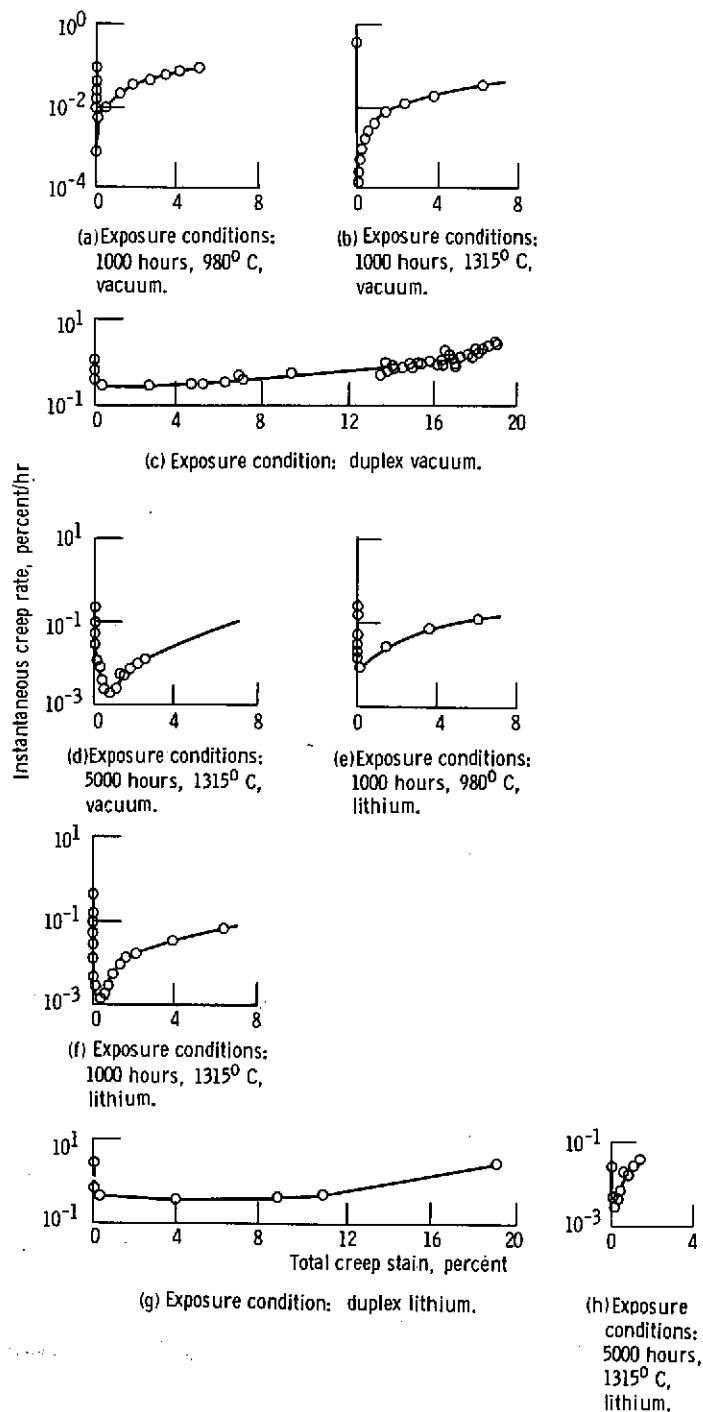


Figure 10. - Effect of exposure to lithium and 10^{-7} newton per square meter (10^{-9} torr) vacuum on creep curve shape for T-111 alloy creep tested at 900° C and 345 meganewtons per square meter (50 ksi).